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Effects of HF Treatments on Tensile Strength of Hi-Nicalon Fibers

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February 1998

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Acknowledgments

Thanks are due to Dan Gorican for fiber strength measurements, John Setlock for SEM, Rob Dickerson for TEM, and Darwin Boyd for scanning Auger analysis.

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EFFECTS OF HF TREATMENTS ON TENSILE STRENGTH OF HI-NICALON FIBERS

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SUMMARY

Tensile strengths of as-received Hi-Nicalon fibers and those having a dual BN/SiC surface coating, deposited by chemical vapor deposition, have been measured at room temperature. These fibers were also treated with HF for 24 hr followed by tensile strength measurements. Strengths of uncoated and BN/SiC coated Hi-Nicalon fibers extracted from celsian matrix composites, by dissolving away the matrix in HF for 24 hr, were also determined. The average tensile strength of uncoated Hi-Nicalon was 3.19 ± 0.73 GPa with a Weibull modulus of 5.41. The Hi-Nicalon/BN/SiC fibers showed an average strength of 3.04 ± 0.53 GPa and Weibull modulus of 6.66. After HF treatments, the average strengths of the uncoated and BN/SiC coated Hi-Nicalon fibers were 2.69 ± 0.67 GPa and 2.80 ± 0.53 GPa and the Weibull moduli were 4.93 and 5.96, respectively. The BN/SiC coated fibers extracted from the celsian matrix composite exhibited a strength of 2.38 ± 0.40 GPa and a Weibull modulus of 7.15. The strength of the uncoated Hi-Nicalon fibers in the composite was so severely degraded that they disintegrated into small fragments during extraction with HF. The uncoated fibers probably undergo mechanical surface damage during hot pressing of the composites. Also, the BN layer on the coated fibers acts as a compliant layer which protects the fibers from mechanical damage during composite processing. The elemental composition and thickness of the fiber coatings were determined using scanning Auger analysis. Microstructural analyses of the fibers and the coatings were done by scanning electron microscopy and transmission electron microscopy. Strengths of fibers calculated using average and measured fiber diameters were in good agreement. Thus, the strength of fibers can be evaluated using an average fiber diameter instead of the measured diameter of each filament.

1. INTRODUCTION

Hi-Nicalon fiber (ref. 1) has a desirable combination of tensile strength, elastic modulus, density, and retention of these properties at temperatures up to ~1200 °C. Thus it is suitable for a variety of aerospace applications as a reinforcement in high performance structural composites with ceramic, metal and polymer matrices. The Hi-Nicalon fiber surface needs to be protected with appropriate ceramic coating(s) in order to alleviate chemical reactions with ceramic and metal matrices during composite processing and use and also, to provide a weak fiber-matrix interface for toughened ceramic composites.

In a recent study (ref. 2) it was observed that the $0.75BaO - 0.25SrO - Al_2O_3 - 2SiO_2$ (BSAS) celsian matrix composite reinforced with Hi-Nicalon (uncoated) fibers showed low flexure strength along with catastrophic failure. In contrast, the composite reinforced with BN/SiC coated Hi-Nicalon fibers was strong, tough and showed graceful failure. Fiber push-through tests and microscopic examinations indicated a weak fiber-matrix interface in both composites, and no chemical reaction was observed between the fibers and the matrix. It was postulated that the low strength of the Hi-Nicalon (uncoated)/celsian composite was due to degradation of fiber strength during composite processing.

The primary objective of the present research was to verify the postulate of this earlier study. Another objective was to carry out microstructural and chemical analyses of the fibers and the coatings. Room temperature tensile strengths of the as-received Hi-Nicalon fibers and those coated with a dual BN/SiC surface layer were measured. Strength of fibers which had been treated with 40 percent HF for 24 hr at room temperature were also determined. In order to investigate the fiber strength degradation during composite processing, strength of fibers extracted from celsian matrix composites, by leaching away the oxide matrix in 40 percent HF for 24 hr at room temperature, were also measured. The Weibull statistical parameters were determined for each type of fiber. Elemental compositions and thicknesses of the fiber coatings were determined by scanning Auger analysis. Microstructural analyses of the fibers and the coatings were done by scanning electron microscopy (SEM) and transmission electron microscopy (TEM).

2. MATERIALS

Hi-Nicalon fiber from Nippon Carbon Company was used in the present study. The properties of the as-received fiber are shown in Table I. These fibers contain nanocrystalline (10 to 20 nm) β-SiC along with 30 to 40 atomic% excess carbon which is also reflected in its low elastic modulus. A large variation (10 to 18 μm) in the fiber diameter was observed, although the manufacturer reports an average value of ~14 μm.

The polyvinyl alcohol (PVA) sizing on the Hi-Nicalon fibers was burned off in a Bunsen burner flame. Single filaments were carefully separated from the fiber tow for testing. All the coatings on the fibers were applied by chemical vapor deposition (CVD) using a continuous CVD reactor. The BN coating was deposited at ~1000 °C utilizing a proprietary precursor and was amorphous to partly turbostratic in nature. A thin over-coating of SiC was also applied to the BN-coated fibers. The nominal coating thickness was 0.4 μm of BN and 0.3 μm of SiC.

3. EXPERIMENTAL PROCEDURES

3.1. Electron Microscopy

Surfaces of the uncoated and BN/SiC coated loose Hi-Nicalon fibers were examined using SEM and TEM. For cross-sectional analysis, fibers were mounted in a high temperature epoxy and polished before examination. SEM was performed using a JEOL JSM-840A operating at 15 keV. Fiber cross-sectional thin foils for TEM were prepared using a procedure developed for ceramic fibers which involves epoxy potting, slicing, polishing, dimple grinding, and Ar ion beam milling. A thin carbon coating was evaporated onto the TEM thin foils and SEM specimens for electrical conductivity prior to analysis. The thin foils were examined in a Philips EM400T operating at 120 keV.

3.2. Scanning Auger Analysis

The elemental compositions of the fiber near the surface and of the fiber surface coatings were analyzed (ref. 3) with a scanning Auger microprobe (Fisons Instruments Microlab Model 310-F). The fibers for this analysis were mounted on a stainless steel sample mount by tacking the ends with colloidal graphite. Depth profiling was performed by sequential ion-beam sputtering and Auger analysis. The ion etching was done with 3 keV Argon ions rastered over an ~1 mm^2 area on the specimen. The etch rate in Ta_2O_5 under these conditions was 0.05 nm/s.

Auger electron spectroscopy (AES) analysis was performed using an electron beam current of ~1.5 nA. The beam was rastered over a 2×20 μm area of the fiber with the long axis of the area aligned with the long axis of the fiber. Spectra were acquired in integral (as opposed to derivative) form at a beam energy of 2 keV and depth profiles were created by plotting peak areas against ion etch time. The atomic concentrations were calculated by dividing the peak areas by the spectrometer transmission function and the sensitivity factors for each peak, then scaling the results to total 100 percent. The sensitivity factors were derived from spectra of ion etched Ag, Si, B, Ti, SiC, BN, and TiO_2 standards. The sensitivity factors used for each element should not be trusted to better than ± 20 percent. The depth scale is from the Ta_2O_5 calibration and no attempt has been made to adjust for the actual etch rate for each material. Only the fibers with a smooth surface coating, rather than those having thick and rough coating morphologies, were used for Auger analysis.

3.3. Tensile Strength Measurement

Room temperature tensile strengths of the individual filaments were measured in ambient atmosphere with a screw driven Micropull fiber test frame equipped with pneumatic grips. A 1000 g load cell, Sensotec model 34, was used. Measurements were carried out at a constant crosshead speed of 1.261 mm/min (0.05 in./min). A single filament was mounted on a 20 lb paper tab using Hardman extra fast setting epoxy. The side portions of the tab were cut with a hot wire just before application of the load producing a fiber gage length of 2.54 cm (1 in.). Twenty filaments of each type of fiber were tested. For the as-received fibers, actual diameter of each filament tested was measured using a Zeiss Axioskop microscope with a Sony DXC-960MD video camera attached to a Boeckeler VIA-150 video measurement system. Magnification ranges from 100X to

800X, depending upon the size of the fiber. Weibull parameters for tensile strength of as-received fibers were calculated using the measured diameters and also an average diameter of 13.5 μm. For all other fibers tested, tensile strengths were calculated based only on an average filament diameter of 13.5 μm.

4. RESULTS AND DISCUSSION

4.1. Microstructure

SEM micrographs showing the surface of as-received, flame desized Hi-Nicalon fibers are presented in figure 1. The fiber surface appears to be fairly smooth and featureless. EDS compositional spectra (fig. 2) taken from the polished cross-section of the fiber indicates the presence of only Si and C along with a small amount of oxygen which is in qualitative agreement with the manufacturer's data (Table I). The surface of HF-treated Hi-Nicalon fibers is also fairly smooth and featureless as seen from the SEM micrograph in figure 3. SEM micrographs from the surface and polished cross-section of the BN/SiC coated Hi-Nicalon fibers are given in figures 4(a) and (b), respectively. The coating on most of the fibers is smooth and uniform whereas some fibers have a quite granular coating. The BN coating is often nodular as can be seen in figure 5. The nodules in the BN layer remain or, often, are enhanced by the subsequent overcoating of SiC. The number density of the nodules varies considerably from filament to filament, but is more consistent along an individual fiber. Within a tow, the fibers on the outside had thicker and more granular coatings than those on the inside. Overall, the coating thickness was reasonably uniform. Generally, the BN coating thickness was 0.2 to 0.4 μm, but could be much higher at the nodules. The total coating thickness on a fiber could be as high as 7 μm when a granular cluster is attached (fig. 5). Typically, the SiC coating thickness varied between 0.2 to 0.3 μm. Sharp contrast is observed between the fiber, BN coating, and SiC coating in polished cross-sections (fig. 6). The microstructure of these coated fibers is quite similar to those of BN/SiC coated HPZ ceramic fibers (ref. 3).

TEM analysis of the loose, BN/SiC coated Hi-Nicalon fibers in cross-section revealed four distinct layers in the BN coating (fig. 7). While there was no detectable variation in composition between the layers, there was a subtle variation in crystallographic structure. All four layers were turbostratic in structure, but layers 1 and 3 (counting from the fiber outward) were more directional. The basic structural units of the BN are more aligned parallel to the fiber surface, giving sharper cusps in microelectron diffraction patterns (fig. 8). The SiC layer on the as-coated fibers typically ion milled away before electron transparent thin regions were obtained, but appeared to be adherent to the BN layers.

After treatment with HF, the BN/SiC surface coating was cracked, and spalled off at some places on the fibers, as seen in the SEM micrographs in figure 9. SEM micrographs showing surfaces of the uncoated and BN/SiC coated fibers extracted from the celsian composite by dissolving away the matrix in HF are given in figures 10 and 11, respectively. Matrix particles are still sticking to many of the fibers and cracks in the coating are observed on those fibers having a thick granular coating.

4.2. Scanning Auger Analysis

Elemental composition depth profiles, as obtained from scanning Auger analysis of as-received, but desized, Hi-Nicalon fibers and those having BN/SiC surface coatings are shown in figures 12(a) and (b), respectively. Depth profiles of the uncoated and coated fibers, after treatment with HF, are also presented in figures 12(c) and (d), respectively. The elemental composition (atomic%) of the as-received fiber is ~42 percent Si and 56 percent C with C/Si atomic ratio of 1.33. A trace amount of oxygen is also detected. These results are in good agreement with the fiber composition supplied by the manufacturer (Table I). The composition of the HF treated Hi-Nicalon fiber is found to be ~43 percent Si, ~54 percent C, and a trace of oxygen. The Hi-Nicalon/BN/SiC fiber has ~130 nm thick layer of Si-rich SiC followed by ~500 nm thick layer of B-rich BN. However, the coatings thickness was nonuniform from filament to filament as seen from the SEM micrographs (figs. 4 and 5) taken from surface and cross-section of the fibers. The fibers on the outside of a tow had thicker and more granular coatings than those on the inside. The BN coating is contaminated with oxygen and contains a high concentration of carbon. In fact, the concentration of carbon is larger than those of boron and nitrogen. The results for the HF treated Hi-Nicalon/BN/SiC fiber are quite similar to those for the untreated fiber. However, a trace amount of F is also detected in the BN layer of the treated fiber.

4.3. Tensile Strength

The strength of ceramic fibers is determined by the statistical distribution of flaws in the material. The tensile strength of ceramic fibers is generally analyzed on the basis of the well known Weibull statistics (ref. 4). An excellent review of the subject was provided by Van der Zwaag (ref. 5). The Weibull modulus describes the distribution of strength in materials which fail at defects according to the weakest link statistics. The probability of survival of a material is given by the empirical equation:

$$P_s = 1 - P(\sigma) = \exp[-V\{(\sigma - \sigma_u)/\sigma_o\}^m] \quad (1)$$

where P_s is the survival probability ($P(\sigma) = 1- P_s$ is the failure probability) of an individual fiber at an applied stress of σ, V is the fiber volume, σ_u is the stress below which failure never occurs, σ_o is the scale parameter, and m is the shape or flaw dispersion parameter. For a given material, the Weibull modulus m and σ_o are constant. Assuming $\sigma_u = 0$ and uniform fiber diameter along the length, L, corresponding to the volume, V, equation (1) can be rearranged as:

$$\ln \ln (1/P_s) = \ln \ln [1/(1 - P(\sigma))] = m \ln\sigma + \text{constant} \quad (2)$$

The experimental data are ranked in ascending order of strength values and the cumulative probability $P(\sigma_i)$ is assigned as

$$P(\sigma_i) = i/(1 + N) \quad (3)$$

where i is the rank of the tested fiber in the ranked strength tabulation and N is the total number of fibers tested. A least-squares linear regression analysis is then applied to a plot of $[\ln\ln 1/P_s]$ vs. $\ln(\sigma)$ whose slope is the Weibull modulus m.

The $\ln \ln (1/P_s)$ versus $\ln \sigma$ Weibull probability plots for room temperature tensile strength of uncoated Hi-Nicalon fibers using measured and average diameters of the filaments are presented in figure 13 and the values of Weibull parameters obtained from linear regression analysis are summarized in Table II. The tensile strength of as-received Hi-Nicalon fiber is 3.30 ± 0.57 GPa and the Weibull modulus, m, is 7.03 using measured diameter for each filament tested. The average value of fiber diameter was found to be 13.5 μm. Using this average diameter, values of fiber tensile strength and m were calculated to be 3.19 ± 0.73 GPa and 5.41, respectively. The manufacturer's information data sheet (ref. 1) reports a value of 2.8 GPa for the room temperature tensile strength of these fibers. The strengths of fibers obtained from measured and average fiber diameters are in good agreement. Thus, an average fiber diameter, instead of the measured diameter of each fiber, can be used in determining fiber strength. The Weibull modulus calculated from measured and average filament diameters are in reasonable agreement. Similar results have also been reported very recently by Petry et al. (ref. 6) from tensile strength measurements of Nicalon and alumina/yttrium aluminum garnet eutectic fibers. The Weibull parameters for the tensile strengths of other fibers tested in the present study were evaluated using an average filament diameter of 13.5 μm.

Weibull plots for the uncoated Hi-Nicalon fibers and those having a dual BN/SiC surface coating are shown in figure 14. Similar Weibull plots for the uncoated and BN/SiC coated Hi-Nicalon fibers which have been treated with 40 percent HF for 24 hr and those extracted from the celsian matrix composite are given in figure 15. Values of Weibull parameters obtained from linear regression analysis for various fibers are summarized in Table II. The BN/SiC coated Hi-Nicalon fibers show an average tensile strength of 3.04 ± 0.53 GPa with m value of 6.66 indicating no strength degradation during fiber coating by CVD. After HF treatment, both the as-received and the BN/SiC coated fibers show strength degradation of ~8 to 15 percent. The tensile strength of the BN/SiC coated fibers extracted from the celsian matrix composite by dissolving away the matrix in HF has degraded to 2.38 ± 0.40 GPa with an m value of 7.15. Exposure of the fibers to high temperature during hot pressing of the composites could probably account for this strength degradation. In contrast, the fibers extracted from the Hi-Nicalon (uncoated)/celsian composite were so weak that they disintegrated into small pieces during extraction with HF. The strength of these uncoated fibers probably degrade due to mechanical surface damage during composite hot pressing. The BN layer in the BN/SiC coated fibers acts as a compliant layer protecting the fibers from mechanical damage during composite processing. SEM micrographs showing typical fracture surfaces of uncoated and BN/SiC coated Hi-Nicalon fibers in the celsian matrix composite are shown in figure 16. Fracture surfaces of the two fibers are seen to be quite different. Uncoated Hi-Nicalon fibers extracted from lithium aluminosilicate (LAS) glass-ceramic matrix composites (ref. 7), which were hot pressed at 1360 °C for 40 min, showed only 20 to 25 percent reduction in

tensile strength. This is because the LAS composites were densified for a short time just above the matrix melting point making use of the viscous flow of glass.

SUMMARY OF RESULTS

Room temperature tensile strengths of as-received Hi-Nicalon fibers and those having a CVD dual BN/SiC surface coating have been determined. Tensile strengths of these fibers following treatment with HF for 24 hr have also been measured. Room temperature tensile strengths of uncoated and BN/SiC coated Hi-Nicalon fibers extracted from celsian matrix composites, by leaching away the matrix in HF for 24 hr, were also determined. The average tensile strength of as-received Hi-Nicalon was 3.19 ± 0.73 GPa with a Weibull modulus of 5.41. Also, tensile strengths of uncoated fibers calculated using average and measured fiber diameters were in good agreement. The BN/SiC coated Hi-Nicalon fibers showed an average strength of 3.04 ± 0.53 GPa and Weibull modulus of 6.66. After HF treatments, the average strengths of the uncoated and BN/SiC coated Hi-Nicalon fibers were 2.69 ± 0.67 GPa and 2.80 ± 0.53 GPa and the Weibull modulus were 4.93 and 5.96, respectively. The BN/SiC coated fibers extracted from celsian matrix composite exhibited a strength of 2.38 ± 0.40 GPa and Weibull modulus of 7.15. The strength of the uncoated Hi-Nicalon fibers in the composite had been so severely degraded that they disintegrated into small pieces during extraction with HF. This strength degradation is probably caused by mechanical surface damage to the uncoated fibers during hot pressing of the composites. The elemental composition and thickness of the fiber coatings were determined using scanning Auger analysis. Microstructural analyses of the fibers and the coatings were done by SEM and TEM. The BN coating, consisting of four distinct layers, was amorphous to partly turbostratic and contaminated heavily with carbon and somewhat with oxygen. Silicon carbide coating was crystalline, nonstoichiometric, nonuniform and sometime flaky.

CONCLUSIONS

The BN coating in the BN/SiC coated Hi-Nicalon fibers acts as a compliant layer protecting the fibers from mechanical damage during composite processing. Tensile strength of fibers can be evaluated using an average diameter instead of the measured diameter of each filament. This should circumvent the amount of time and effort spent in the measurement of diameter of individual filaments.

ACKNOWLEDGMENTS

Thanks are due to Dan Gorican for fiber strength measurements, John Setlock for SEM, Rob Dickerson for TEM, and Darwin Boyd for scanning Auger analysis.

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Table I. Properties of Hi-Nicalon Fibers*

Property	Value
Density, g/cm^3	2.74
Diameter, μm	14
Filaments/tow	500
Denier, g/9000m	1800
Tensile strength, GPa	2.8
Elastic modulus, GPa	269
Thermal expansion coefficient, $10^{-6}/K$	3.5 (25-500 °C)
Specific heat, J/g-K	0.67 (25 °C) 1.17 (500 °C)
Thermal conductivity, W/m-K	7.77 (25 °C) 10.1 (500 °C)
Electrical resistivity, Ω-cm	1.4
Chemical composition, wt%	63.7 Si, 35.8 C, 0.5 O
C/Si, atomic ratio	1.3-1.4
Temperature capability, °C	1200

*Data from Nippon Carbon Company.

Table II. Weibull Parameters for Room Temperature Tensile Strength of Hi-Nicalon Fibers
[Gage length = 2.54 cm; Crosshead speed = 1.261 mm/min]

Fiber treatment	Fiber diameter, μm	Average strength, GPa	Standard deviation, GPa	Weibull modulus, m
As-received, flame desized Hi-Nicalon	Actual*	3.30	0.57	7.03
As-received, flame desized Hi-Nicalon	13.5 (average)	3.19	0.73	5.41
As-received, flame desized Hi-Nicalon; treated with 40 percent HF, 24 hr	13.5 (average)	2.69	0.67	4.93
BN-SiC coated Hi-Nicalon	13.5 (average)	3.04	0.53	6.66
BN-SiC coated Hi-Nicalon; treated with 40 percent HF, 24 hr	13.5 (average)	2.80	0.53	5.96
BN-SiC coated Hi-Nicalon, extracted from celsian composite by dissolving away matrix in 40 percent HF, 24 hr	13.5 (average)	2.38	0.40	7.15

*Actual fiber diameter measured for each filament tested. Twenty filaments tested for each fiber type.

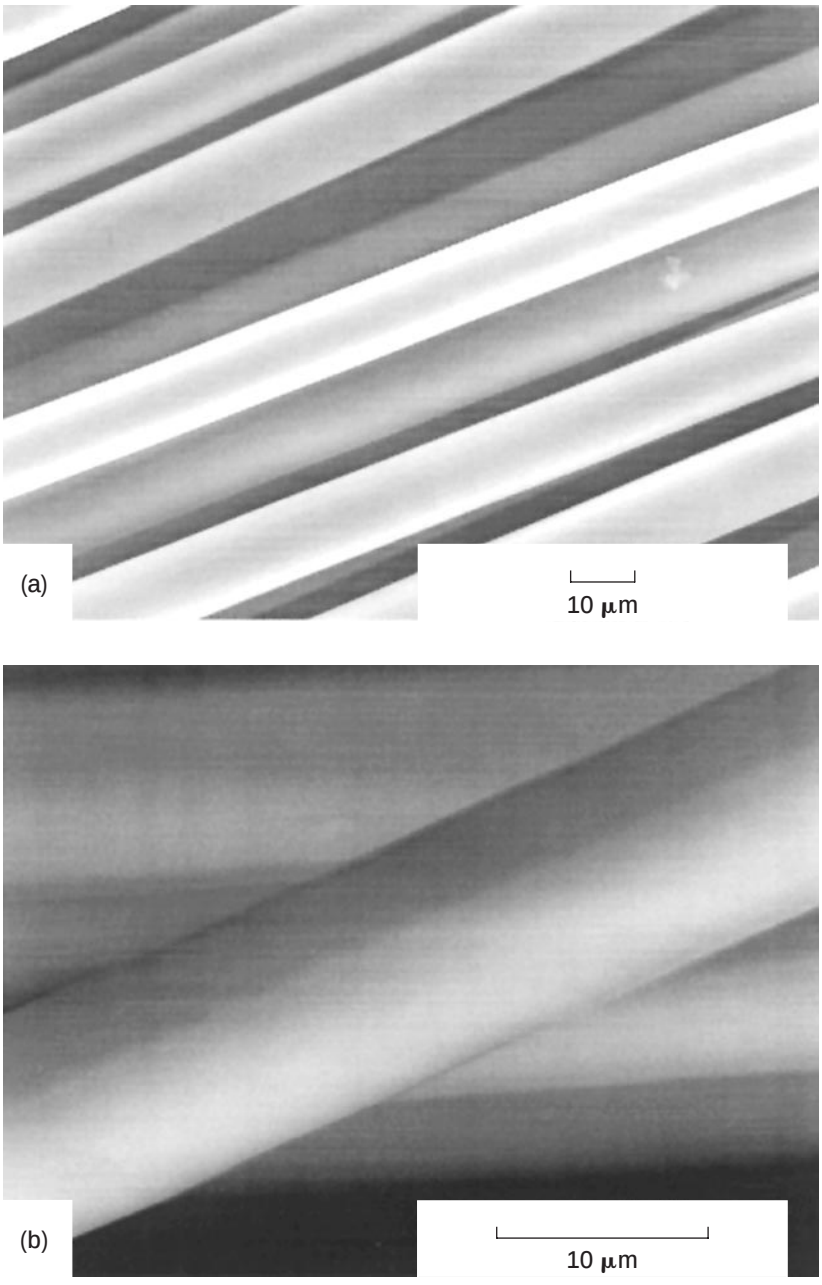


Figure 1.—SEM micrographs showing surface of desized Hi-Nicalon fibers.

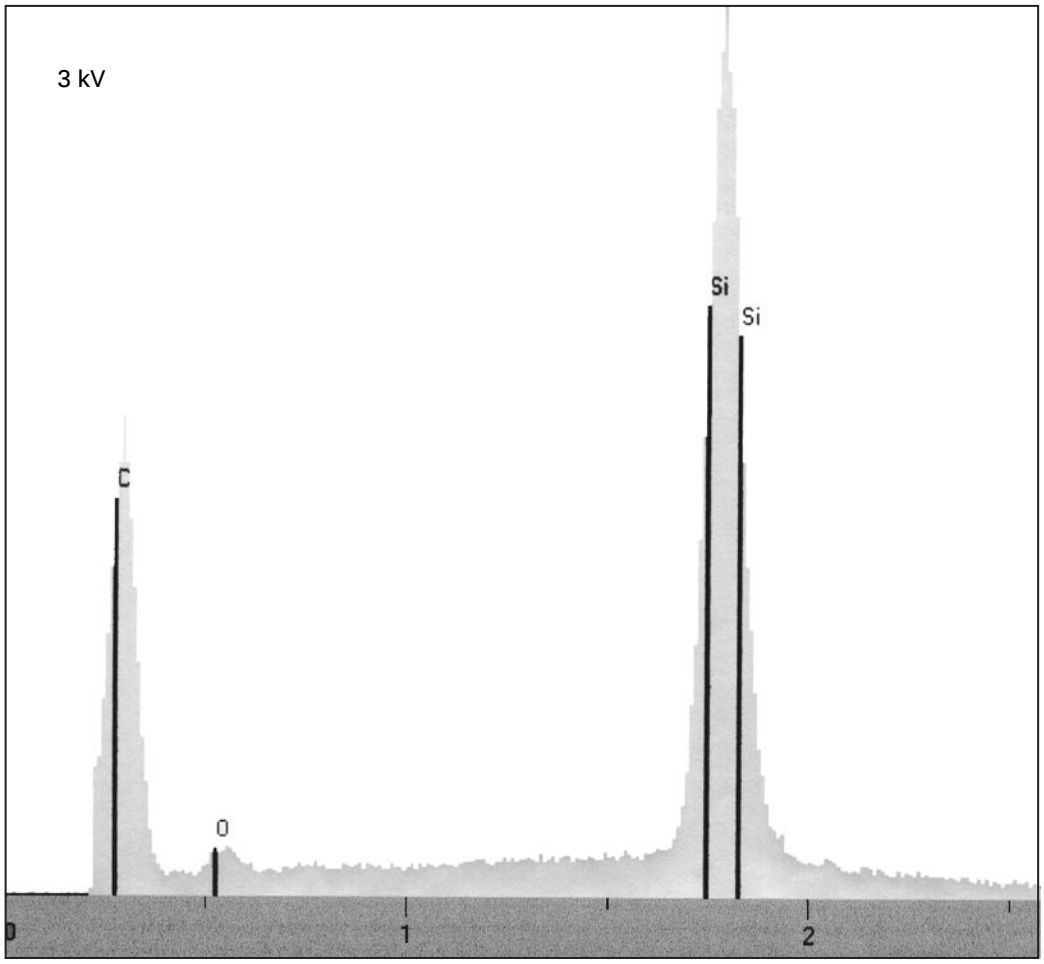


Figure 2.—EDS spectra from the cross-section of desized Hi-Nicalon fiber at 3 kV.

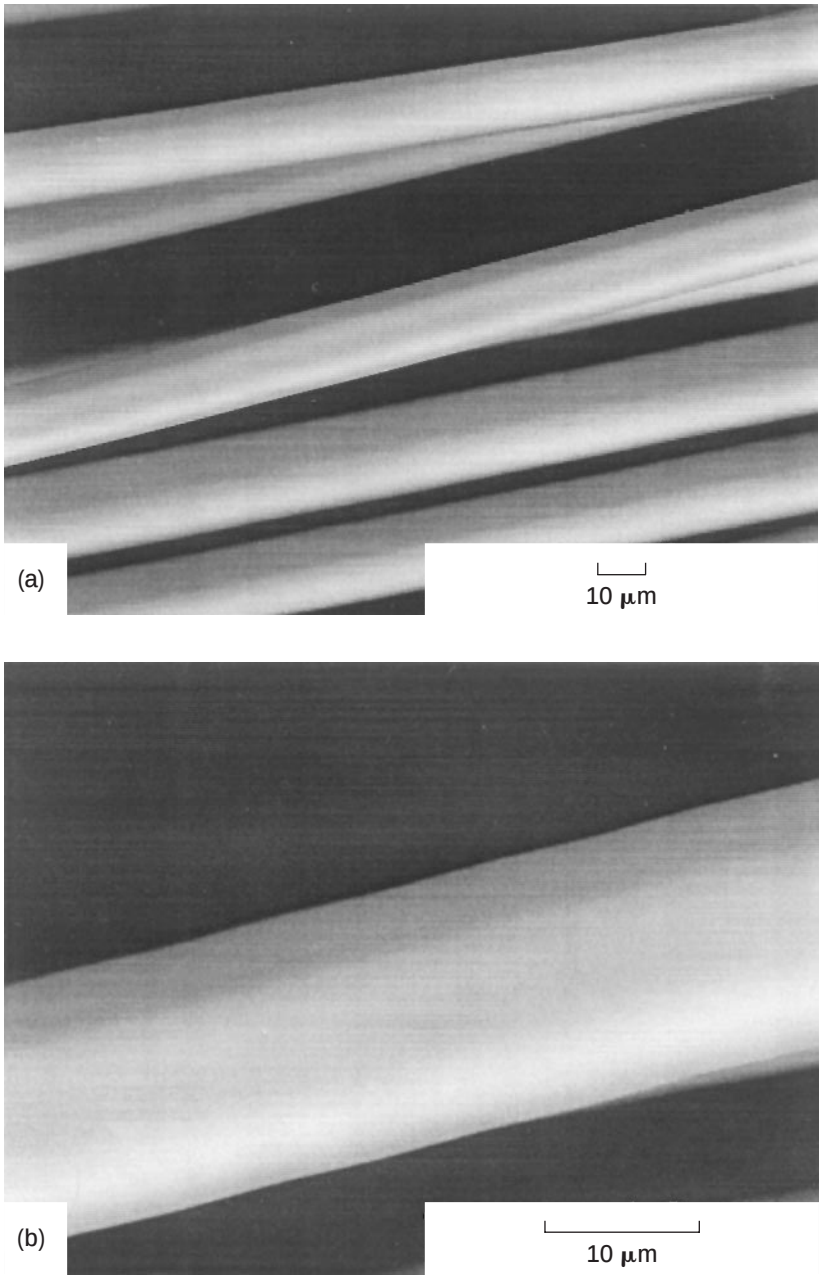


Figure 3.—SEM micrographs showing surface of Hi-Nicalon fiber after treatment with HF.

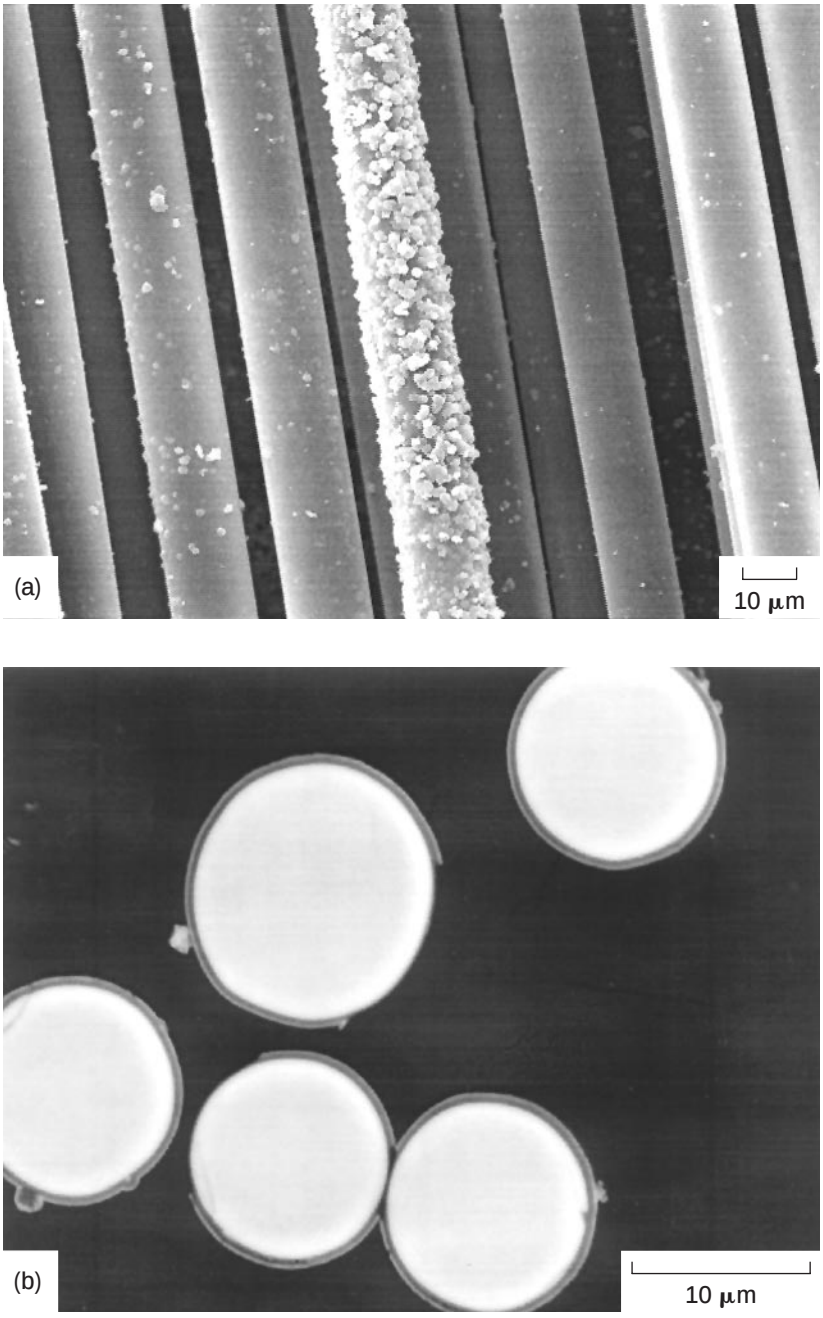


Figure 4.—SEM micrographs. (a) Surface. (b) Polished cross-section of BN/SiC coated Hi-Nicalon fibers.

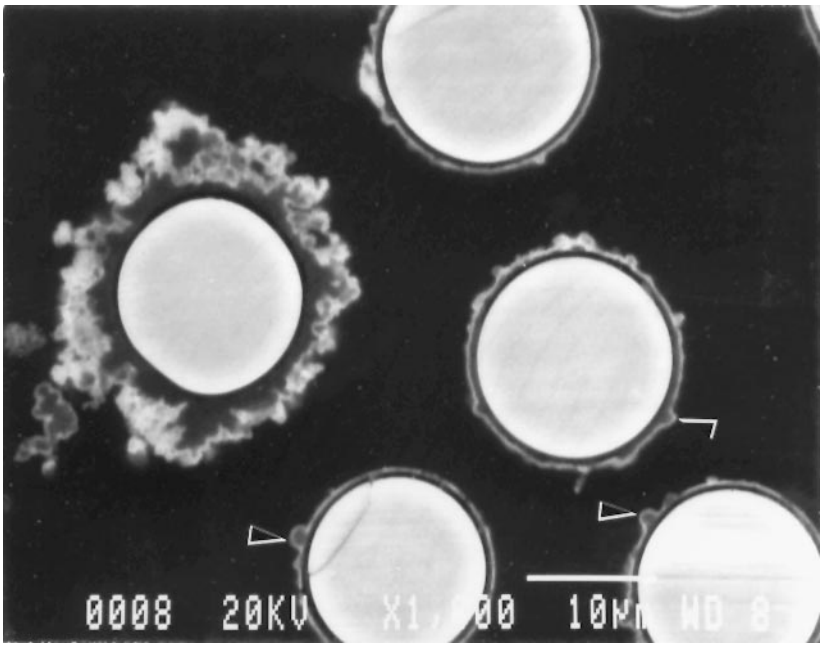


Figure 5.—SEM micrograph from polished cross-section of BN/SiC coated Hi-Nicalon fibers showing nodular coating. Some of the nodules are indicated by arrows.

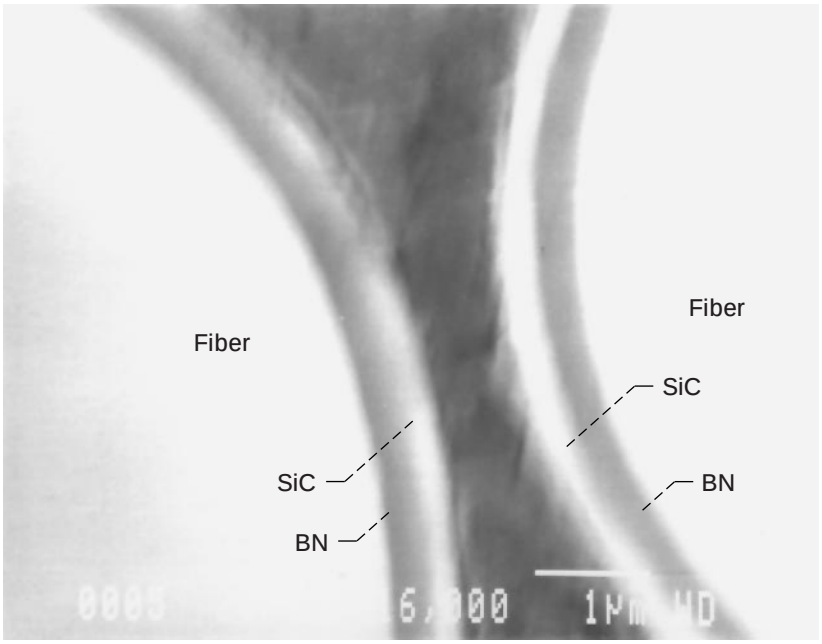


Figure 6.—SEM micrograph at high magnification from polished cross-section of BN/SiC coated Hi-Nicalon fibers showing sharp contrast between the fiber and the coatings.

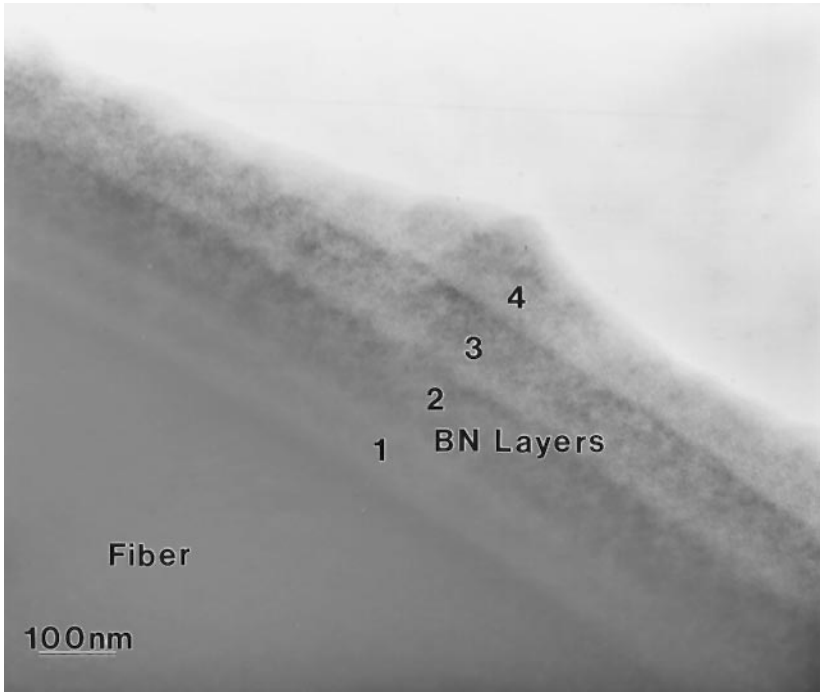


Figure 7.—TEM bright field image showing cross-section of the as-received BN/SiC coated Hi-Nicalon fiber; four distinct sub-layers are visible within the BN coating.

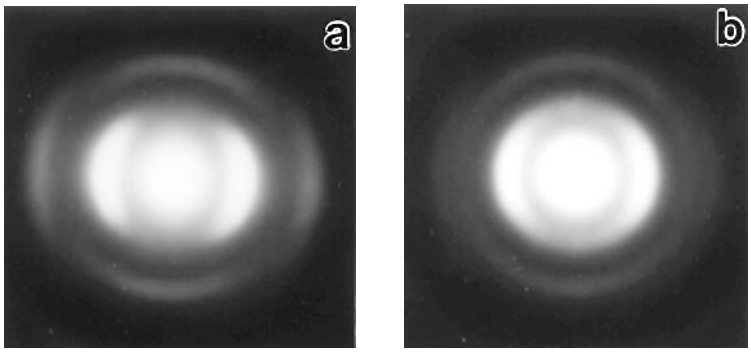


Figure 8.—Microelectron diffraction patterns typical for BN layers in BN/SiC coated Hi-Nicalon fibers. (a) Layers 1 and 3. (b) Layers 2 and 4.

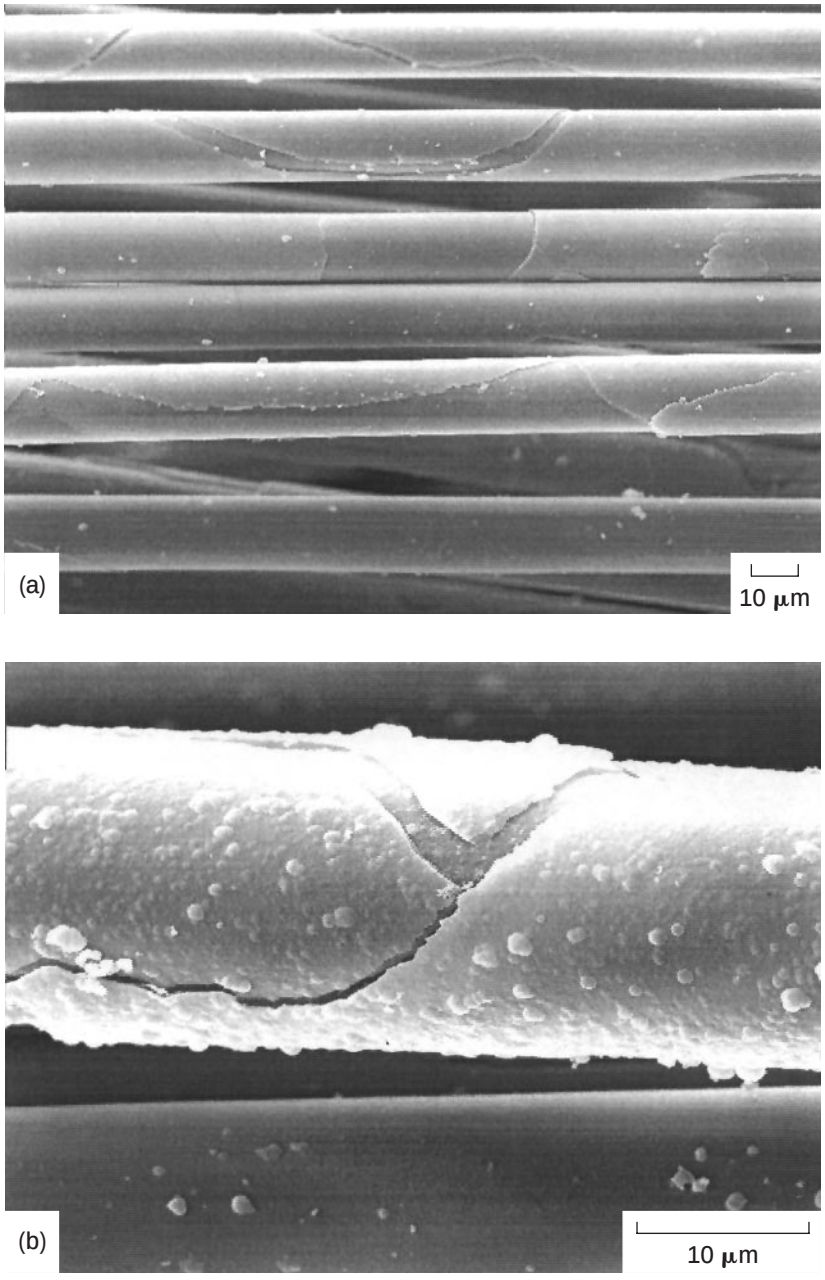


Figure 9.—SEM micrographs showing surface of BN/SiC coated Hi-Nicalon after treatment with HF.

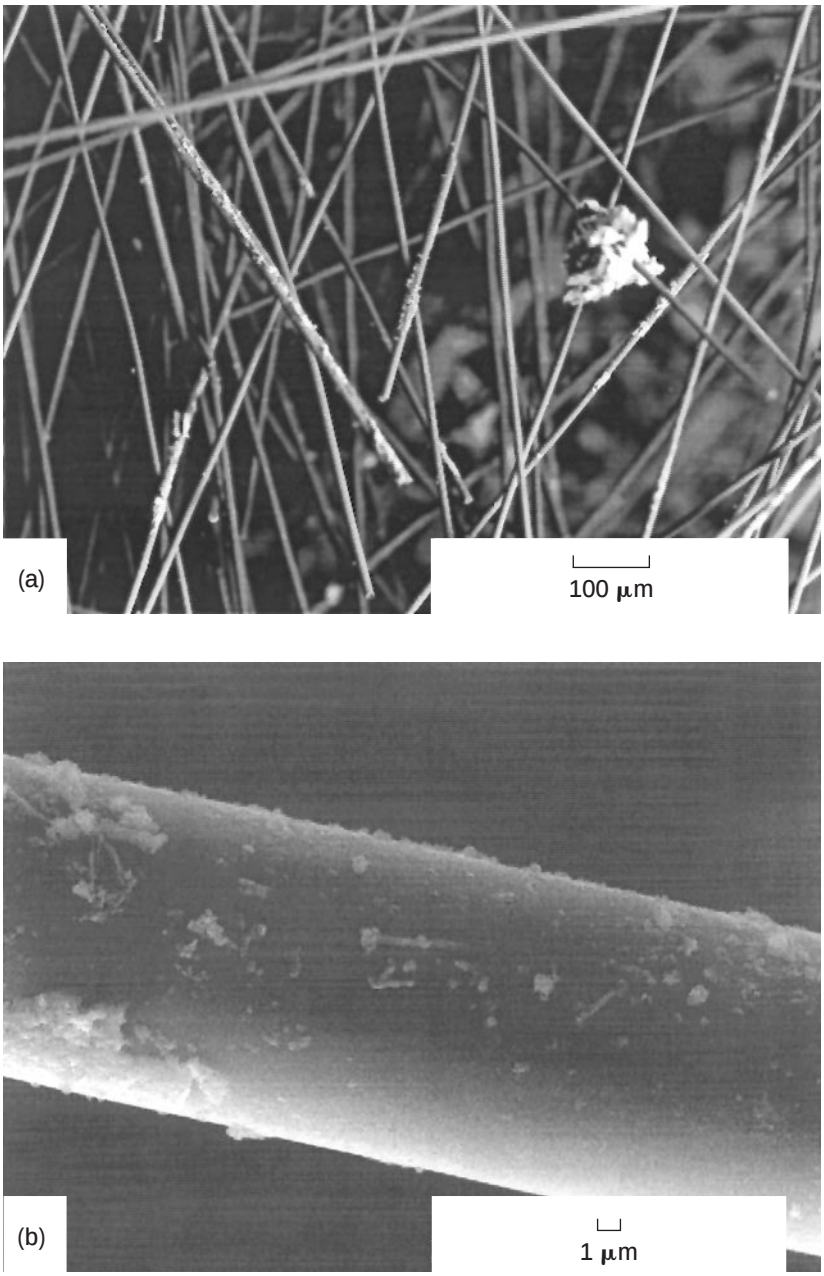


Figure 10.—SEM micrographs showing surface of uncoated Hi-Nicalon fibers extracted from celsian matrix composite by dissolving away the matrix in HF.

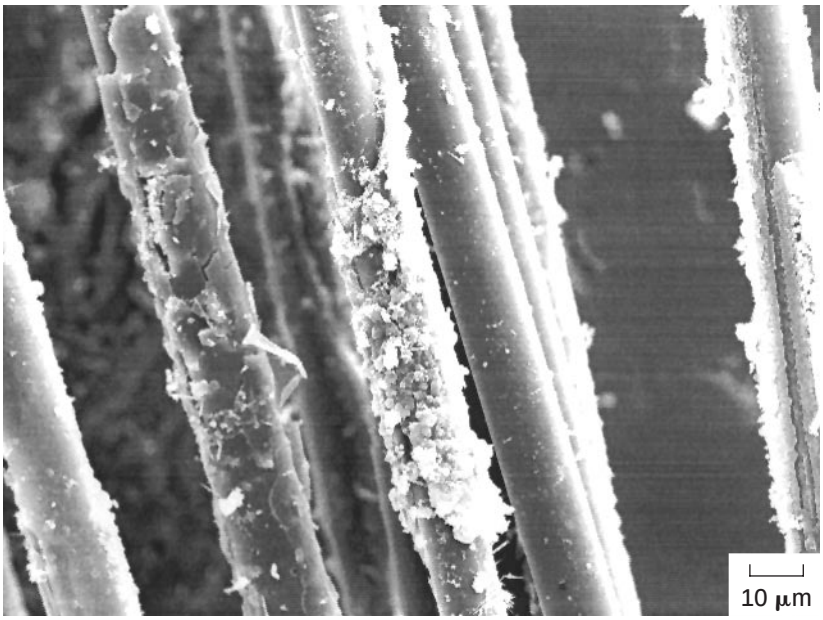


Figure 11.—SEM micrograph showing surface of BN/SiC coated Hi-Nicalon fibers extracted from celsian matrix composite by dissolving away the matrix in HF.

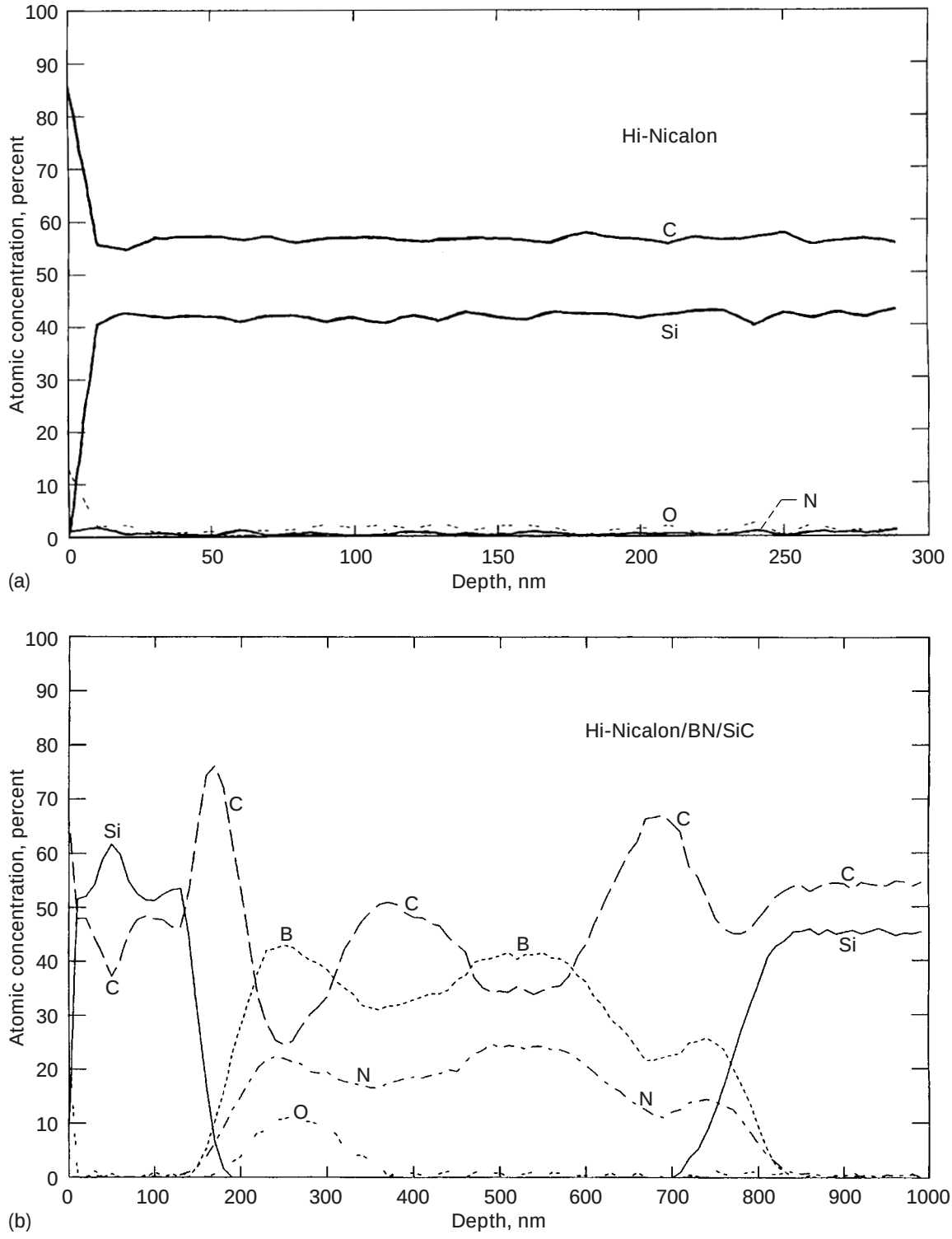
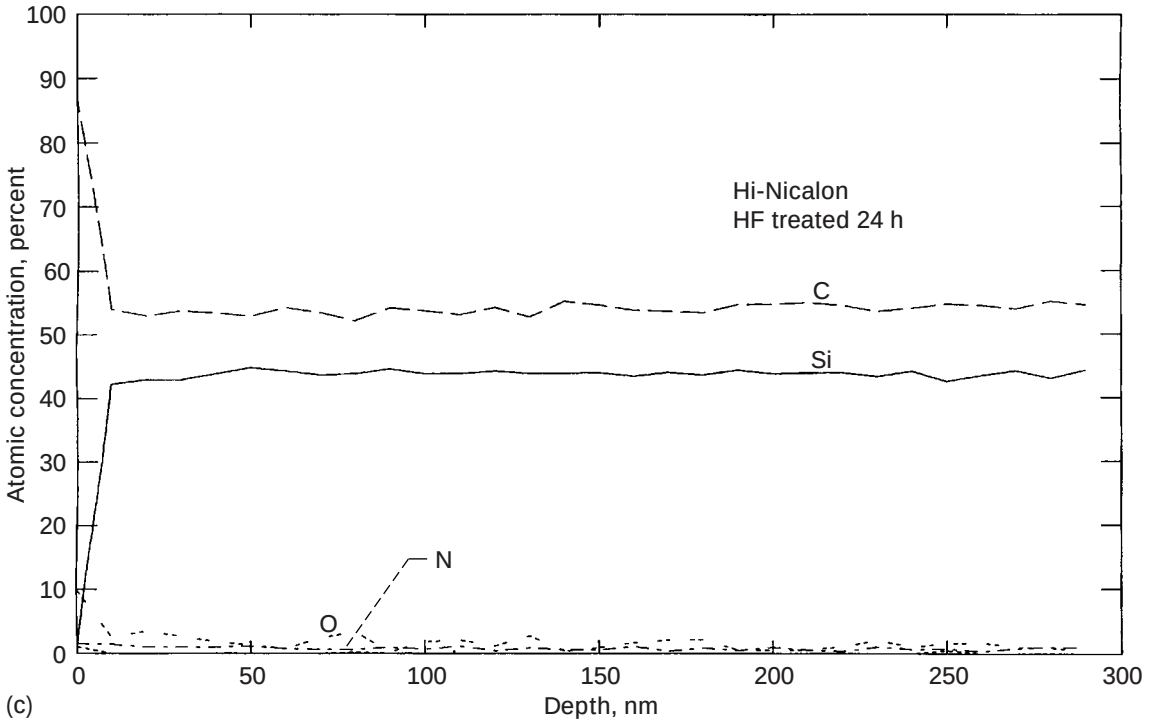


Figure 12.—Surface depth profiles of various elements obtained using scanning Auger microprobe. (a) Hi-Nicalon. (b) Hi-Nicalon/BN/SiC. (c) HF treated Hi-Nicalon. (d) HF treated Hi-Nicalon/BN/SiC fibers.



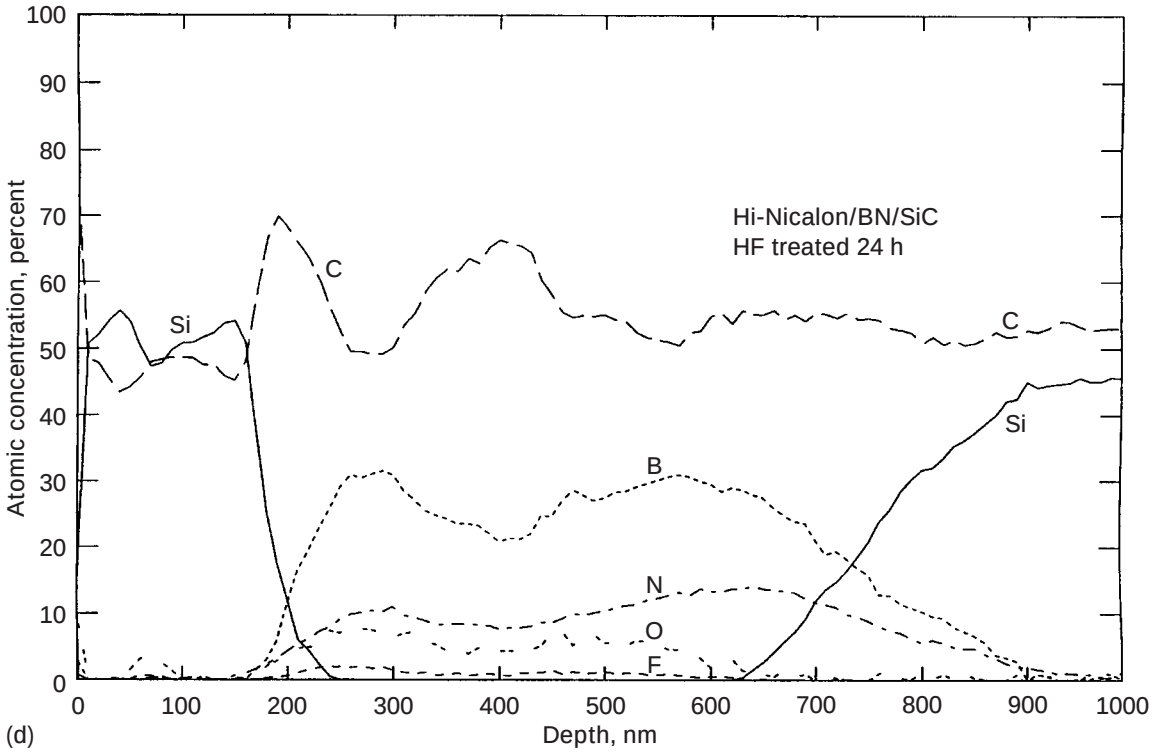


Figure 12.—Concluded. (c) HF treated Hi-Nicalon. (d) HF treated Hi-Nicalon/BN/SiC fibers.

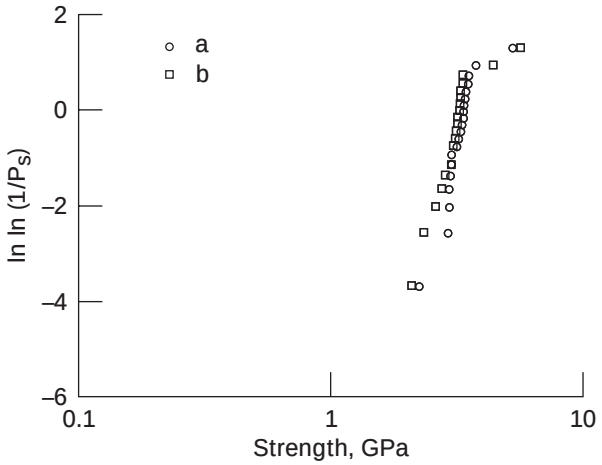


Figure 13.—Weibull probability plots for room temperature tensile strengths of Hi-Nicalon fibers using (a) Measured, (b) Average diameter of the filaments.

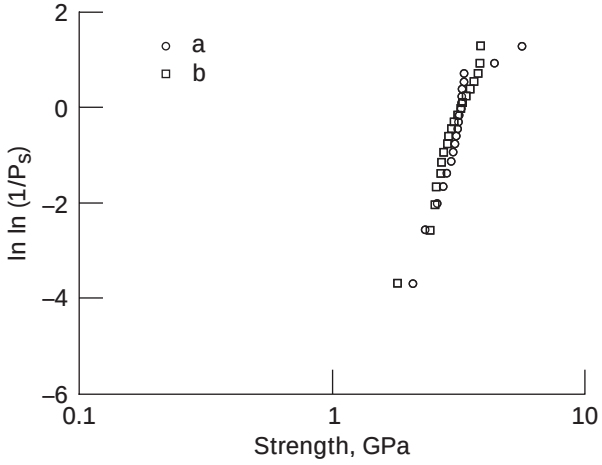


Figure 14.—Weibull probability plots for room temperature tensile strengths of fibers. (a) Hi-Nicalon. (b) BN/SiC coated Hi-Nicalon.

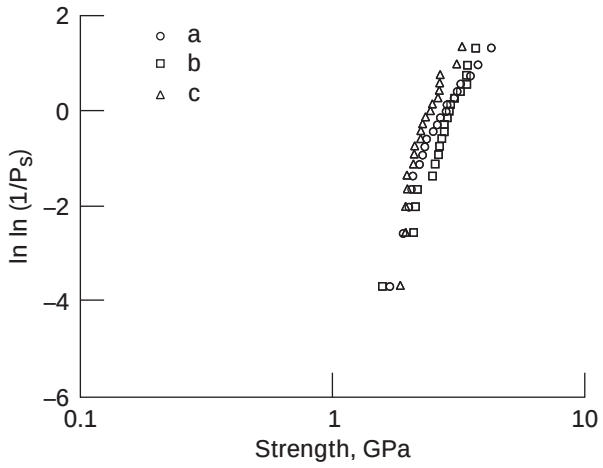


Figure 15.—Weibull probability plots for room temperature tensile strengths of chemically treated fibers. (a) HF treated Hi-Nicalon. (b) BN/SiC coated Hi-Nicalon treated with HF. (c) BN/SiC coated Hi-Nicalon extracted from celsian matrix composite by leaching away matrix in HF.

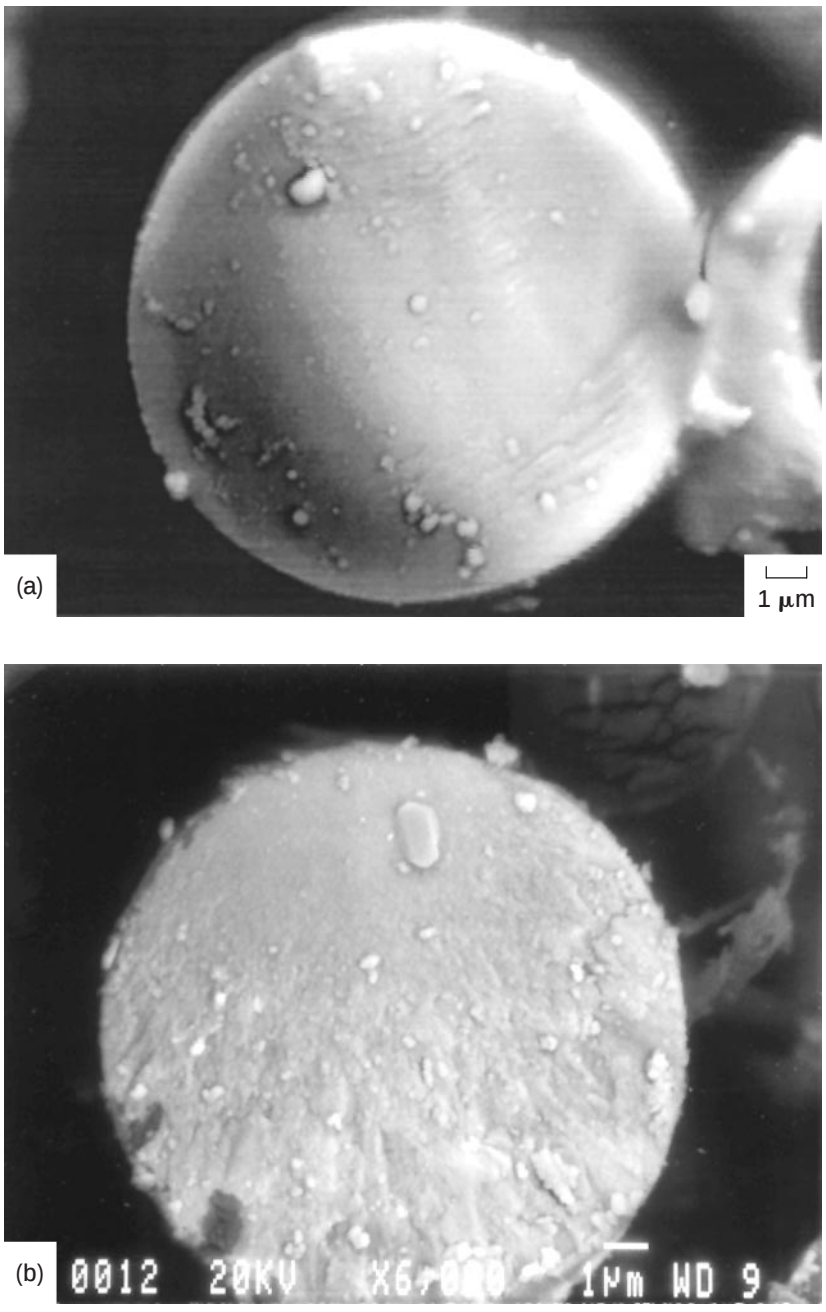


Figure 16.—SEM micrographs showing typical fracture surfaces. (a) As-received, (b) BN/SiC coated Hi-Nicalon fibers in celsian matrix composites.

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13. ABSTRACT ***(Maximum 200 words)***

Tensile strengths of as-received Hi-Nicalon fibers and those having a dual BN/SiC surface coating, deposited by chemical vapor deposition, have been measured at room temperature. These fibers were also treated with HF for 24 h followed by tensile strength measurements. Strengths of uncoated and BN/SiC coated Hi-Nicalon fibers extracted from celsian matrix composites, by dissolving away the matrix in HF for 24 h, were also determined. The average tensile strength of uncoated Hi-Nicalon was 3.19 ± 0.73 GPa with a Weibull modulus of 5.41. The Hi-Nicalon/BN/SiC fibers showed an average strength of 3.04 ± 0.53 GPa and Weibull modulus of 6.66. After HF treatments, the average strengths of the uncoated and BN/SiC coated Hi-Nicalon fibers were 2.69 ± 0.67 GPa and 2.80 ± 0.53 GPa and the Weibull moduli were 4.93 and 5.96, respectively. The BN/SiC coated fibers extracted from the celsian matrix composite exhibited a strength of 2.38 ± 0.40 GPa and a Weibull modulus of 7.15. The strength of the uncoated Hi-Nicalon fibers in the composite was so severely degraded that they disintegrated into small fragments during extraction with HF. The uncoated fibers probably undergo mechanical surface damage during hot pressing of the composites. Also, the BN layer on the coated fibers acts as a compliant layer which protects the fibers from mechanical damage during composite processing. The elemental composition and thickness of the fiber coatings were determined using scanning Auger analysis. Microstructural analyses of the fibers and the coatings were done by scanning electron microscopy and transmission electron microscopy. Strengths of fibers calculated using average and measured fiber diameters were in good agreement. Thus, the strength of fibers can be evaluated using an average fiber diameter instead of the measured diameter of each filament.

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